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Improved Synthesis of Intermetal Compounds

The problem:

At the present, most of the reactive compounds in the polycrystalline form are produced using the horizontal Bridgman techniques. However, because of this lengthy processing, the cost of these compounds is high.

The solution:

An improved method in crystal growth has been developed by using liquid encapsulation techniques. Three GaAs synthesis runs, yielding approximately 140 gm/run, were successfully completed under a molten B_2O_3 encapsulant with ambient gas pressure up to 6.2×10^7 dynes/cm² (900 psig). The developed technique permits the synthesis of materials from the constituent elements whose vapor pressures are high at the temperature at which they react spontaneously.

How it's done:

The GaAs synthesis is accomplished in the high-pressure furnace through the following heating cycle:

(a) By the use of the degas susceptor, arsenic oxide is vaporized at 300°C and 10^{-4} Torr for 12 hours;

(b) The B_2O_3 is then passed over the arsenic oxide charge at 425°C and 1.7×10^6 dynes/cm² (25 psi) for 2 to 4 hours;

(c) Under increased temperature and pressure of 525°C and 5.4×10^7 dynes/cm² (800 psi), respectively, the B_2O_3 is driven into the voids in the charge for 1 to 2 hours;

(d) The elements are reacted to form the GaAs melt under 1450°C and 5.4×10^7 dynes/cm² for 1 hour;

(e) For an additional 1/2 hour, the melt is homogenized by freezing it to 1200°C and then raising the temperature to 1450°C again under the constant pressure of 5.4×10^7 dynes/cm²;

(f) The pressure is now lowered to 1.4×10^6 dynes/cm² (20 psi) and the compound is cooled at room temperature for 1/2 hour.

During steps (a) and (b), the degas susceptor is positioned low within the rf heating coil to induce an inverted steep axial temperature gradient allowing the B_2O_3 to flow without excessive heating of the unreacted As. After step (c), the susceptor is raised within the rf coil and retained in this position through the remainder of the synthesis. The described technique may also be useful for synthesis of GaP.

Patent status:

Title to this invention has been waived under the provisions of the National Aeronautics and Space Act [42 U.S.C. 2457 (f)], to the Arthur D. Little, Inc., Cambridge, Massachusetts, 02140.

Source: J. S. Haggerty and J. Wenckus of
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